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Towards quantitative three-dimensional characterisation of InAs quantum dots

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InAs quantum dots (QDs) grown on InP or InGaAsP are used for optical communication applications operating in the 1.3 – 1.55 µm wavelength range. It is generally understood that the optical properties of such QDs are highly dependent on their three-dimensional structural and chemical profiles. Whilst conventional transmission electron microscopy (TEM) techniques can be used to study capped QDs in plan-view or cross-sectional geometries, the resulting images can provide ambiguous information about their three-dimensional properties. Here, we describe an approach for investigating the applicability of both high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) tomography and atom probe tomography (APT) to the study of surface and buried InAs/InGaAsP QDs grown by metal organic vapour phase epitaxy (MOVPE).

Electron tomography was carried out in an FEI Titan TEM instrument operated at 300 kV. TEM specimens were prepared in plan-view geometry using mechanical grinding, polishing and Ar ion milling. Both original HAADF STEM images and final tomographic reconstruction of surface QDs suggest an elongated hexagonal shape for the bases of the QDs (Figure 1). The elongation direction was determined to be [110], using selected area electron diffraction and atomic force microscopy. The HAADF STEM images also suggest that surface QDs have a double-terraced geometry, with steeper facets around their bases and shallower facets close to their tops. This geometry is consistent with a theoretical model of InAs QDs formed on an InGaAs substrate that is lattice matched to InP [1] shown in Figure 1(b). Despite the large inner detector semi-angle used (approximately 50 mrad), strong diffraction effects were present in the original tilt series of HAADF STEM images, resulting in departure from the projection requirement for electron tomography, which states that the recorded intensity should be a monotonic function of a property of the object [2]. These diffraction effects are likely to be associated with diffraction and may lead to artefacts in the tomographic reconstruction. The same tomographic analysis was applied to a buried InAs/InGaAsP QD (Figure 1(d) and (e)). The buried QD appears to be elongated along the [110] direction, although not as strongly as the surface QD. Similarly, the faceting that is clearly visible in both the original HAADF STEM images and the final reconstruction of the surface QD, is not as pronounced for the buried QD. This difference may result from chemical intermixing between the buried QD and the capping material during overgrowth.

A limiting constraint in STEM tomography of thin film specimens is the limited tilt range available before the specimen becomes too thick for imaging. This limitation can, in principle, be overcome by fabricating needle-shaped specimens using focused ion beam (FIB) milling, in order to allow unlimited tilting without significant increase in projected specimen thickness. However, FIB milling can introduce considerable damage into III-V semiconductors, including amorphisation and Ga ion implantation [3]. We have fabricated needle-shaped specimens that are 100 nm in diameter, using reactive ion etching, selective wet etching and critical point drying in plan-view geometry (Figure 2). The choice of a plan-view geometry for the needles means that each specimen will contain several QDs. The needles can either be detached from the substrate by cleaving (Figure 2(b)) or lifted out and mounted onto suitable grids using a micro-manipulator in the FIB with minimal additional damage (Figure 2(c)). Significantly, in addition to their suitability for electron tomography, these specimens can be used for APT, for which needle-shaped specimens with sharp tips (narrower than 100 nm) are required. Our ongoing experiments involve the application of both HAADF STEM tomography and APT to the same QD, in order to better understand its morphology and composition. A comparison between reconstructions obtained using both techniques will also assist in the evaluation and mitigation of potential artefacts that are present when using each technique.

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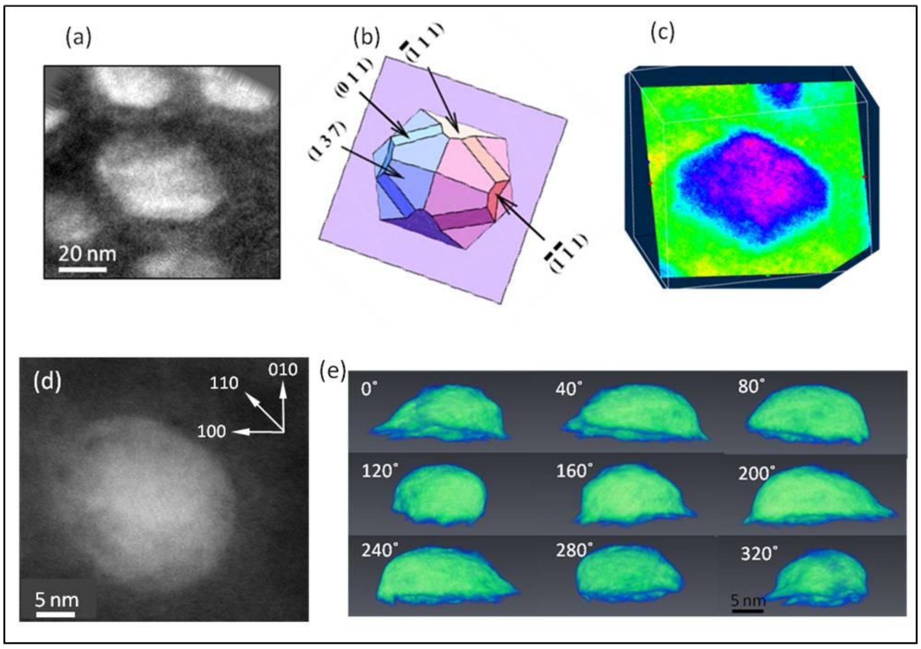

Figure 1. (a) HAADF STEM image of a surface InAs QD on an InGaAsP substrate that is lattice matched to InP. (b) Theoretical energetically favourable shape of InAs QDs on an InGaAs substrate lattice matched to InP, taken from [4]. (c) Tomographic reconstruction of a surface QD in (a) from 67 HAADF STEM images taken at 2˚ intervals over the tilt range ± 66˚ using the SIRT algorithm. (d) HAADF STEM image of a buried InAs/InGaAsP QD in plan-view geometry viewed along the [001] orientation. (e) Tomography reconstruction of the QD in (d) from 61 images obtained at 2˚ intervals over a ± 60˚ tilt range using the SIRT algorithm (15 iterations).

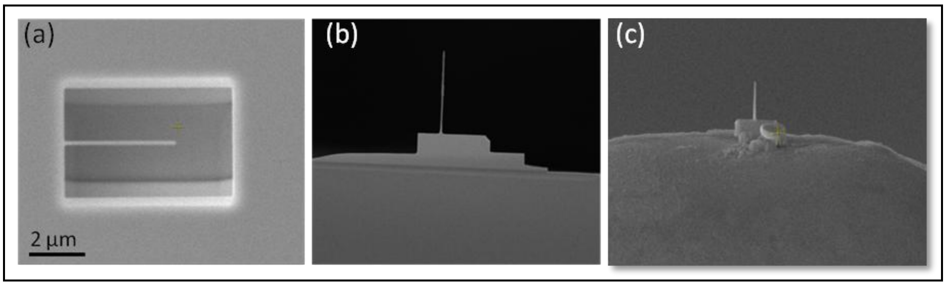

Figure 2. (a) Scanning electron microscopy images of needle-shaped specimens fabricated using reactive ion etching, selective wet etching. The needle specimens can be (b) detached from substrates by cleaving or (c) lifted out and mounted onto a suitable grid.